

Surface sampling & analysis: Examples from NIOSH work

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Examples for Pb, Be & Metals

1. Handwipe disclosing method for the presence of lead (qualitative)
2. Determination of trace beryllium in wipe samples (quantitative)
3. Microvacuum sampling (performance data)

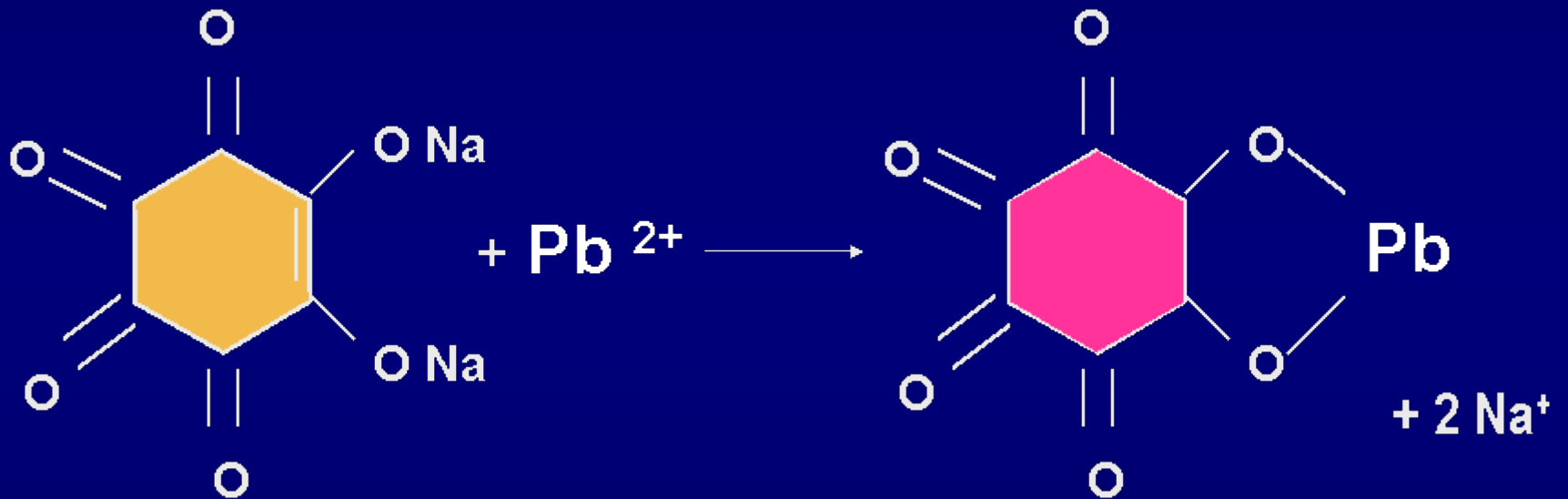


HANDWIPE DISCLOSING METHOD FOR THE PRESENCE OF LEAD*



*[US Pat. 6,248,593]

Sodium Rhodizonate – Lead Colorimetric Reaction



Sodium rhodizonate
(yellow / orange, pH < 7)

Lead – rhodizonate adduct
(pink / red)

HANDWIPE DISCLOSING METHOD FOR THE PRESENCE OF LEAD

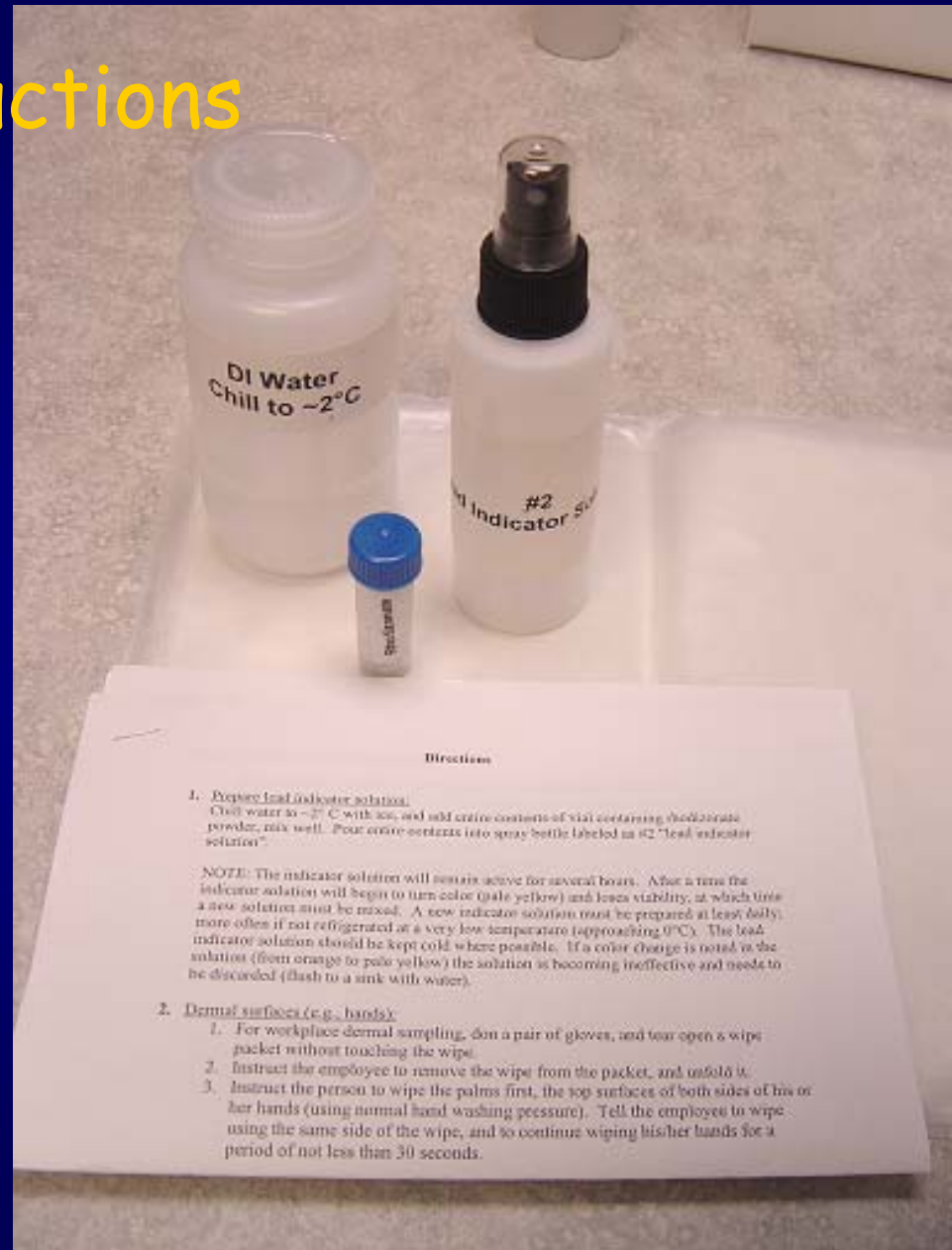
The kit includes:

- 1 instruction sheet
- 1 pre-weighed vial
of rhodizonate powder
- 12 handwipes
(10 samples and 2 blanks)
- 10 pairs of gloves
- 2 pre-labeled spray bottles
- 1 bottle of 105 mL DI water
- 12 50-mL sample collection tubes
- 10 sheets of pre-cut wax paper



1. Read the Instructions

Handwipe disclosing
method for lead –
Commercial product:
"Full Disclosure"



2. Prepare the Pb indicator solution



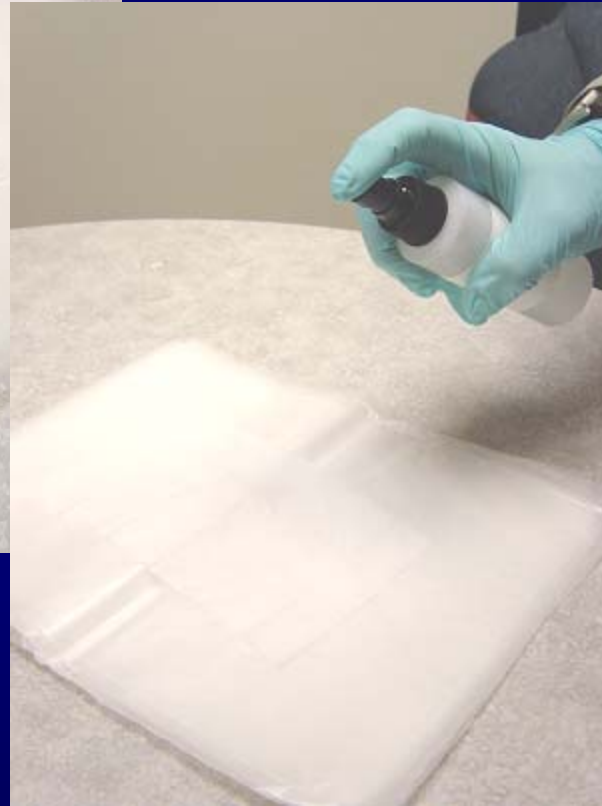
Handwipe disclosing
method for lead, cont'd.

(NIOSH Method 9105)



3. Wipe hands for 30 seconds
(use ASTM E1792 wipes)

Handwipe disclosing
method for lead, cont'd.



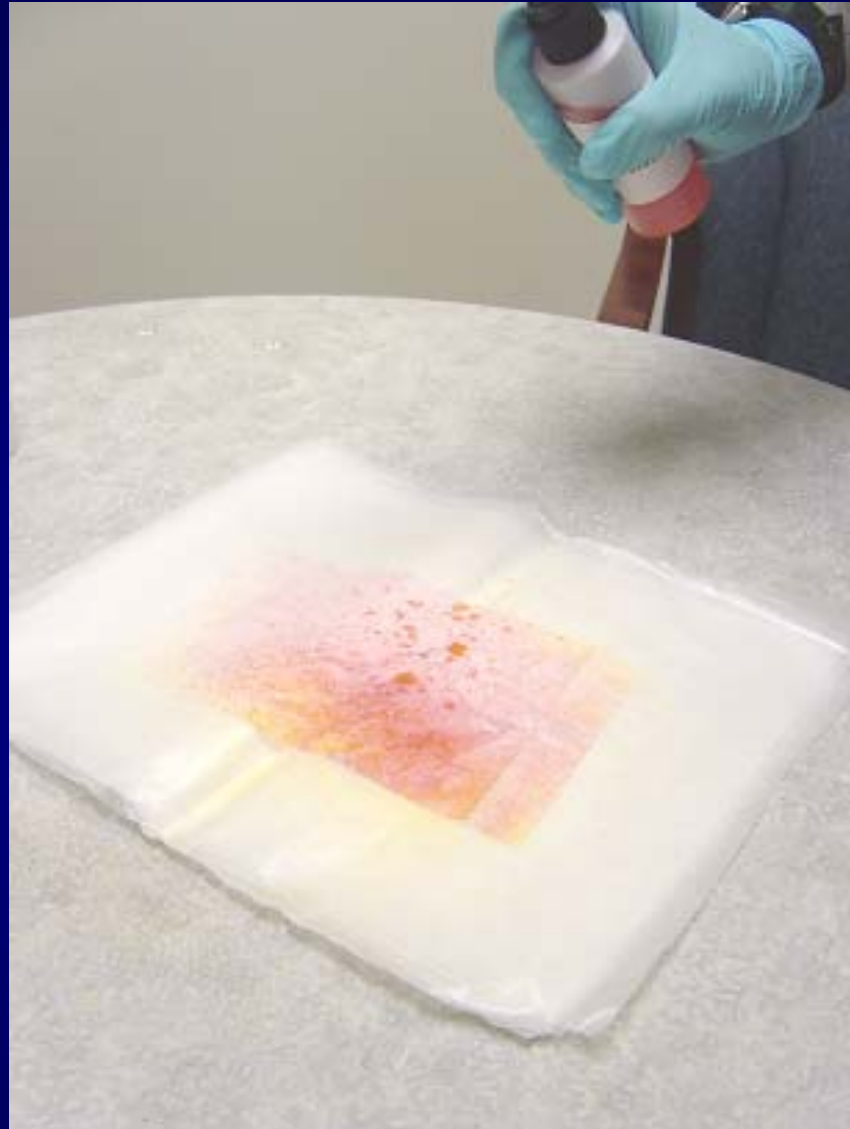
4. Spray 3 pumps of extraction solution
(soln. #1) onto center of wipe

Handwipe disclosing
method for lead, cont'd.



5. Spray 2-3 pumps of the disclosing solution (bottle #2) onto the center of the wipe

Handwipe disclosing
method for lead, cont'd.



6. The presence of Pb is disclosed
if the sample turns a pink to red color

Negative control

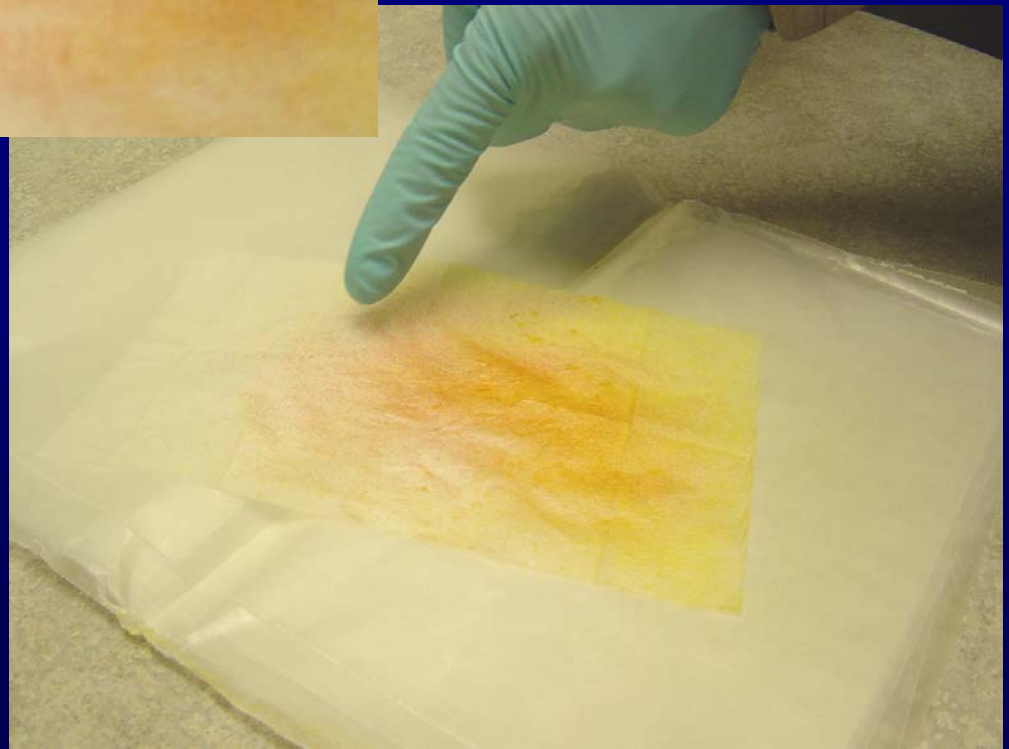
Positive Sample





Handwipe disclosing
method for lead, cont'd.

The method is
sensitive
and specific
for lead



Handwipe disclosing
method for lead, cont'd.



Can be also be used to disclose the
presence of lead on hard surfaces, e.g.,
Floors & Window Sills (pre-clearance),
Shoes (take-home Pb), Car Interiors...

Wet wipe sampling of lead dust – Performance data (ASTM D6966)

Collection efficiency of Pb in dust from smooth surfaces (RTI, 1990s):

75-80% (1st wiping); to >90% (3rd wiping)

Collection efficiency of PbO dust from hands (NIOSH, 2000s):

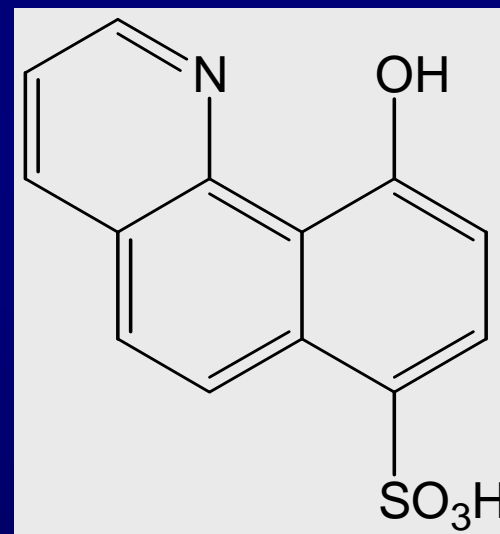
55-60% (1 wipe, 30 sec per pair of hands);
to nearly 80% (3 wipes)



Trace beryllium measurement: New extraction – fluorescence method

[ASTM D7202 / NIOSH 9110]

1. Sample collection using standard methods
2. Extraction of beryllium with dilute ammonium bifluoride, $(\text{NH}_4)\text{HF}_2$
3. Ultra-trace fluorescence measurement of beryllium with high quantum yield fluorophore (LOD <0.001 $\mu\text{g Be/sample}$)



Hydroxybenzoquinoline sulfonic acid (HBQS)

Trace beryllium measurement by extraction/ fluorescence method* – Performance data

<i>Sample / media (n=no. of samples)</i>	<i>Extraction method</i>	<i>Mean % recovery</i>	<i>RSD (%)</i>
Be (n=3)	mechanical	96	3.1
Be/Whatman (n=3)	mechanical	95	4.2
BeO (n=6)	mechanical	86	6.8
BeO (n=3)	heat (85 °C)	95	9.8
BeO/Whatman (n=15)	mechanical	82	5.6
BeO/Whatman (n=6)	heat (85 °C)	96	6.2

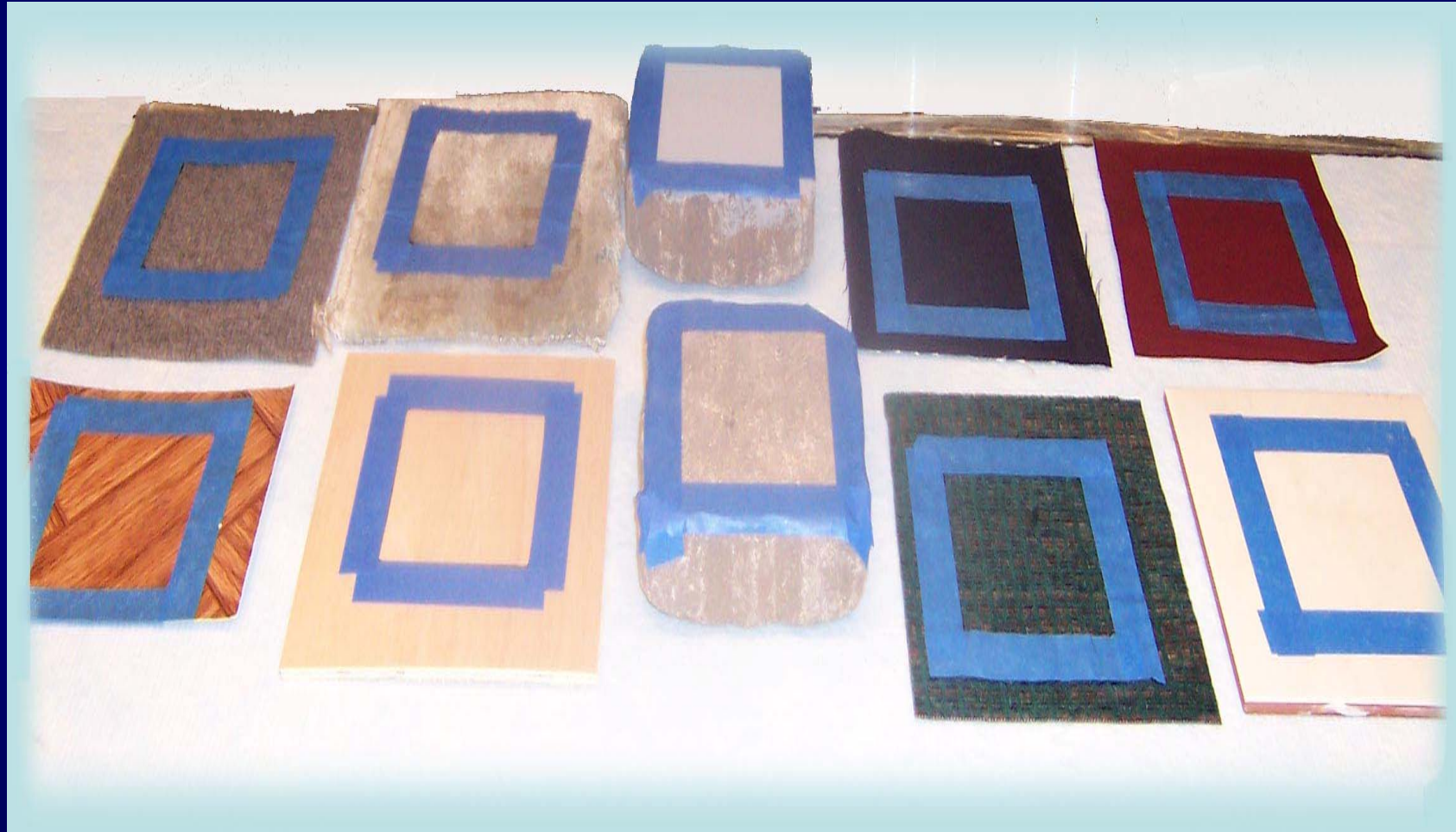
**[Agrawal et al., JEM, 2006; Ashley et al., ACA, 2007]*

Dry wipe sampling of beryllium – Performance data* (ASTM D7296)

Sampling Media	% Recovery (RSD, %)
Wet PVA wipe	86 (7)
Dry PVA wipe	16 (54)
Wet cellulose filter	106 (9)
Dry cellulose filter	43 (25)
Wet smear tab	64 (13)
Dry smear tab	14 (22)

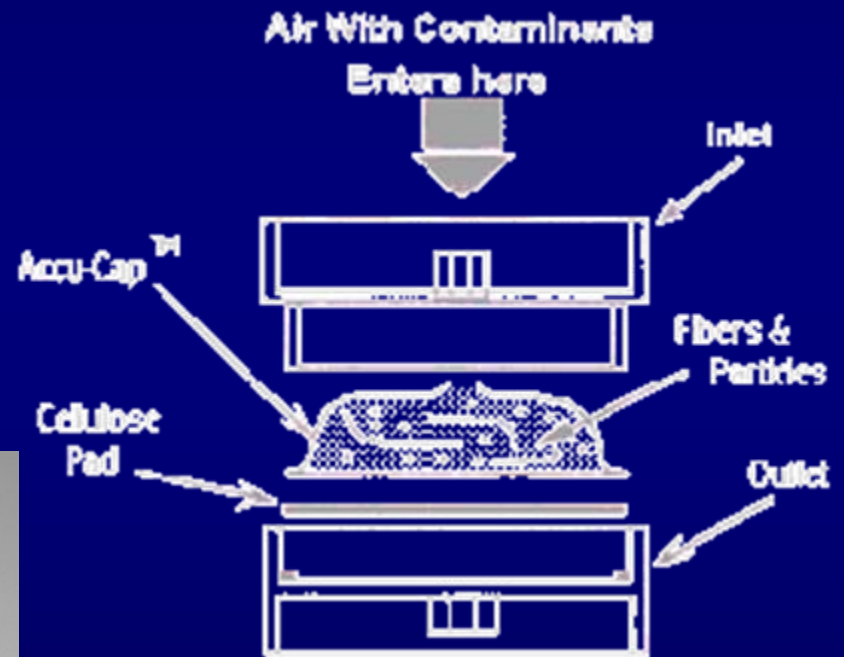
*[*Dufay & Archuleta, JEM, 2006*]

Micro-vacuum sampling: Performance evaluation* (ASTM D7144) – Substrates



*[K. Ashley et al., JOEH, 2007]

PVC inserts (Accu-cap™) for gravimetric analysis



Micro-vacuum sampling – Performance data (soft / rough surfaces)

Substrate material	% Recovery (95% CL's), SRM 1579	% Recovery (95% CL's), SRM 1648	% Recovery (95% CL's), SRM 2583
Industrial carpet	22 (10)	32 (14)	27 (8)
Plush carpet	36 (30)	34 (24)	41 (16)
Car seat material	31 (18)	49 (12)	49 (12)
Denim	45 (17)	37 (13)	55 (21)
Concrete block	64 (210)	69 (37)	87 (72)
Concrete block, painted	33 (14)	45 (21)	43 (26)

Micro-vacuum sampling – Performance data (hard / smooth surfaces)

Substrate material	% Recovery (95% CL's), SRM 1579	% Recovery (95% CL's), SRM 1648	% Recovery (95% CL's), SRM 2583
Glass	59 (11)	43 (10)	50 (14)
Tile	51 (27)	42 (35)	50 (18)
Steel	51 (10)	39 (9)	38 (21)
Linoleum	41 (21)	28 (10)	30 (15)
Vinyl	38 (18)	33 (13)	38 (18)
Wood	34 (19)	33 (10)	49 (23)

Micro-vacuum sampling – Cassette plus collection nozzles (soft / rough surfaces)

Substrate material	Approx. % collected, SRM 1579	Approx. % collected, SRM 1648	Approx. % collected, SRM 2583
Industrial carpet	35	57	50
Plush carpet	59	73	69
Car seat material	55	78	77
Denim	71	81	85
Concrete block	105	113	130
Concrete block, painted	55	72	59

Micro-vacuum sampling – Cassette plus collection nozzles (hard / smooth surfaces)

Substrate material	Approx. % collected, SRM 1579	Approx. % collected, SRM 1648	Approx. % collected, SRM 2583
Glass	87	88	76
Tile	77	88	85
Steel	72	83	71
Linoleum	71	70	56
Vinyl	64	74	65
Wood	55	76	75

Summary - Surface Sampling of Metals

Use standardized protocols and appropriate media to estimate surface contamination of:

Beryllium

Lead

Chromium

Arsenic

Cobalt

Manganese

Cadmium

Silver

Molybdenum

Aluminum

Zinc

Uranium

Mercury

Tin

Nickel

Acknowledgments

Lead handwipe method:

Eric Esswein, CDC/NIOSH

Beryllium fluorescence method:

Anoop Agrawal et al., Berylliant, Inc.

Mark McCleskey et al., Los Alamos Nat'l Lab

Microvacuum sampling method:

Greg Applegate & Tami Wise, CDC/NIOSH